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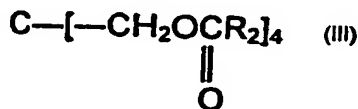
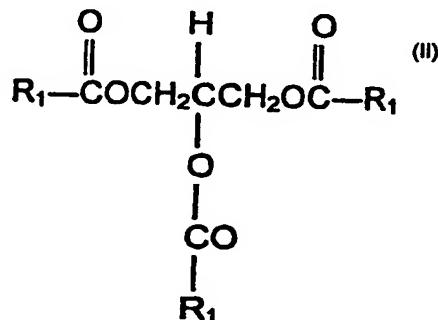
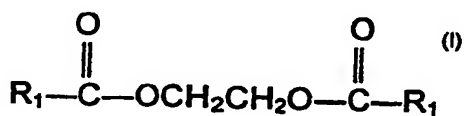
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(54) Title: POLYCARBONATE COMPOSITIONS WITH IMPROVED PROCESSABILITY



(57) Abstract: A polycarbonate composition which comprises an effective amount of at least one lubricant selected from the group consisting of the compounds of formulas (I), (II) and (III), in which R₁ is a mixture of C₂₅-C₃₈ linear alkyl groups and R₂ is a mixture of C₁₅-C₁₇ linear alkyl groups, and mixtures of these compounds.

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Polycarbonate compositions with improved processability.

The present invention relates to polycarbonate (PC) compositions and in particular to polycarbonate compositions having improved processability while maintaining the exceptional mechanical properties of the articles molded therefrom.

Polycarbonates and in particular bisphenol-A polycarbonates constitute a family of polymers which leads to molded articles exhibiting a unique set of mechanical and chemical properties.

More specifically, articles molded from PC compositions exhibit unusually high impact strength even at low temperatures, low moisture absorption, good heat resistance as well as good thermal and oxidative stability in the melt.

Additionally, transparent and self-extinguishing articles can be obtained through molding, for example injection molding, of PC compositions.

For the above reasons, PC compositions are widely used for making molded articles such as medical devices, telephone parts, business machine housings, machinery housings, automobile parts, glazing parts and optical and ophthalmic lenses.

Japanese patent N°50003457 discloses polycarbonates with good pigment dispersibility comprising 0.01-5 parts of montanic acid aliphatic divalent alcohol esters and 0.01-5 parts of pigments, such as TiO_2 .

However, one characteristic of PC compositions is their high melt viscosity. This high melt viscosity of PC compositions renders such compositions difficult to process, in particular the molding thereof. Thus, injection molding of PC compositions is difficult and necessitates high injection pressures.

For this reason, the weight average molecular weight (M_w) of polycarbonates has been in practice limited to values in the range of 15,000 to 65,000, preferably 20,000 to 45,000. Even with these weight average molecular weight ranges, processing of PC compositions is difficult.

In order to improve processing of polymer compositions, additives such as lubricants and processing aids have been added to the polymer compositions, in particular to poly(vinyl chloride) compositions.

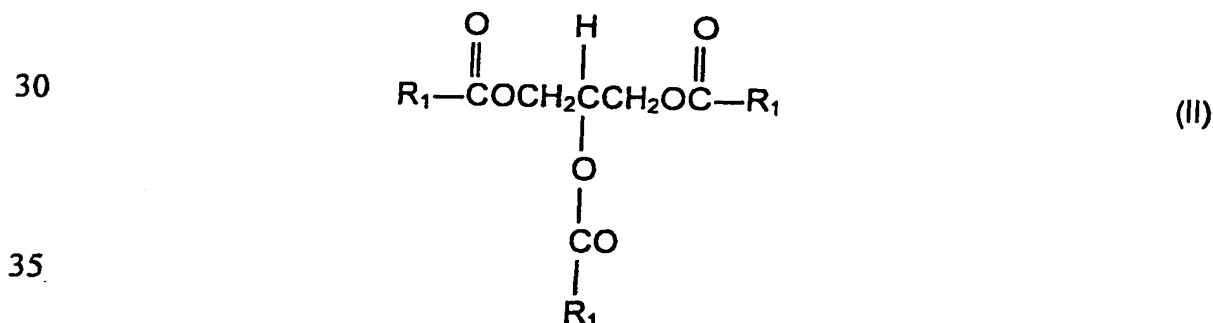
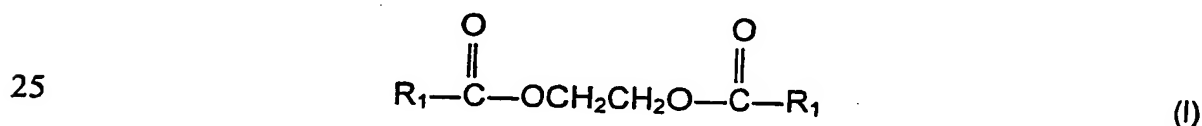
Processing aids are high molecular weight compounds which, in injection molding, promote sticking to the walls of the barrel for improving feeding, improve melt homogeneity and reduce melt fracture.

Lubricants are low molecular weight compounds that reduce melt pressure, decrease adhesion of the polymer or resin composition and improve surface. Lubricants are of two types, "internal" or "external", depending upon their behaviour. Briefly, internal lubricants are compounds that reduce the melt viscosity by acting at the polymer/polymer boundary whereas external lubricants are compounds that reduce the release effect and friction at the polymer/metal boundary, such as mold release agents.

In the formulation of molding polymer compositions the selection of the modifying additives cannot be effected by a simple transposition from one class of polymer compositions to another. For example, effective lubricants or processing aids for poly(vinyl chloride) compositions may be ineffective when used with a different polymer composition such as a polycarbonate composition.

The aim of the present invention is thus to formulate polycarbonate compositions that exhibit improved processability and in particular reduced melt viscosities while still resulting in molded parts having the unique mechanical properties usually obtained in polycarbonate molded parts, such as impact strength.

This goal is achieved according to the present invention by providing a polycarbonate composition which comprises an effective amount of at least one lubricant selected from the group consisting of the compounds of formulas:





5 in which R₁ is a mixture of C₂₈-C₃₈ linear alkyl groups and R₂ is a mixture of C₁₅-C₁₇ linear alkyl groups, and mixtures of these compounds.

The preferred lubricants are mixtures of compounds of formula I (L/MONTAN) such as the product Hoechst Wax E Powder[®] commercialized by Clariant CORP., mixtures of compounds of formula II (M/MONTAN) such as the
10 products Hostalub WE-40[®] commercialized by Clariant Corp. and mixtures of compounds of formula III (L/PETS) such as the product Struktol TRO44[®] OC commercialized by Struktol.

Mixtures of compounds of formulas II and III are also preferred.

The effective amount of lubricant is the necessary amount imparting
15 significant reduction of melt viscosity to the PC composition as compared to the same composition free of lubricant according to the invention. Generally, the amount of lubricant of the invention ranges from 0.01 to 5 % by weight, preferably 0.1 to 2.5 % by weight based on the total weight of polycarbonates present in the composition.

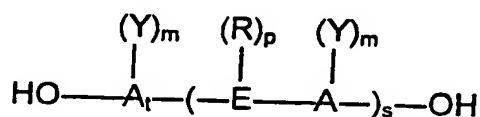
20 The aromatic polycarbonate resins employed in the compositions of the invention are well known. Generally speaking, such carbonate polymers may be typified as possessing recurring structural units of the formula:



wherein D is a divalent aromatic radical of the dihydric phenol employed in the polymerization reaction. Preferably, the polycarbonate polymers used to
30 provide the resinous compositions of the invention have an intrinsic viscosity (as measured in methylene chloride at 25°C ranging from about 0.70 to about 1.4 dl/g). In general, the higher viscosity polycarbonates are preferred. The dihydric phenols which may be employed to provide such aromatic carbonated polymers are mononuclear or polynuclear aromatic compounds, containing as
35 functional groups two hydroxy radicals, each of which is attached directly to a carbon atom of an aromatic nucleus. The preferred polycarbonate resin for use herein is a homopolymer derived from 2,2-bis-(4-hydroxyphenyl)propane and a carbonate precursor.

The aromatic polycarbonates may be manufactured by known processes, such as the methods set forth in U.S. Patent No. 4,018,750 and 4,123,436 where a dihydric phenol is reacted with a carbonate precursor, or by transesterification processes as well as other processes known to those skilled in the art. The preferred method of preparing polycarbonate resins comprises the interfacial polymerization of a dihydric phenol with a carbonate precursor.

Typical dihydric phenols useful in formulating the polycarbonate resins, as described above, may be represented by the general formula:



- in which A is an aromatic group such as phenylene, biphenylene, naphthalene, anthrylene; E may be an alkylene or alkylidene group such as isopropylidene, butylene, butylidene, isobutylidene, amylene, isoamylene, amyridene, isoamyridene, and generally from one to twelve carbon atoms, inclusive. Where E is an alkylene or alkylidene group, it may also consist of two or more alkylene or alkylidene groups connected by a non-alkylene or non-alkylidene groups, such as an aromatic linkage, a tertiary amino linkage, an ether linkage, a carbonyl linkage, or by a sulfur-containing linkage such as sulfide, sulfoxide or sulfone. In addition, E may be a cycloaliphatic group of five to twelve carbon atoms, inclusive (e.g. cyclopentyl, cyclohexyl), or a cycloalkylidene of five to seven carbon atoms, inclusive, such as cyclohexylidene; a sulfur-containing linkage, such as sulfide, sulfoxide or sulfone; an ether linkage; a carbonyl group; a direct bond; or a tertiary nitrogen group. Other groups which E may represent will occur to those skilled in the art. R is hydrogen or a monovalent hydrocarbon group such as alkyl of one to eight carbon atoms, inclusive (methyl, ethyl, propyl); aryl (phenyl, naphthyl); aralkyl (benzyl, ethylphenyl); or cycloaliphatic of five to seven carbon atoms, inclusive (cyclopentyl, cyclohexyl). Y may be an inorganic atom such as chlorine, bromine, fluorine; an organic group such as the nitro group; an organic group such as R above; or an oxy group such as OR, it being only necessary that Y be inert to and unaffected by the reactants and the reaction conditions. The letter m is any whole number from and including zero through the number of positions on A available for substitution; p is any whole number from and including zero through the number

of available positions on E; t is a whole number equal to at least one; and s is any whole number from and including zero to twenty. In the typical dihydric phenol compound represented by formula I above, when more than one Y substituent is present, they may be the same or different. The same is true for the R substituent. Where s is greater than one, E can be the same or different. Where E is a direct bond, the aromatic rings are directly joined with no intervening alkylene or other bridge. The positions of the hydroxyl groups and Y on the aromatic nuclear residues, A, can be varied in the ortho, meta or para positions; and the groupings can be in a vicinal, non-symmetrical or symmetrical relationship, where two or more ring carbon atoms of the aromatic hydrocarbon residue are substituted with Y and a hydroxyl group.

Examples of dihydric phenol compounds that may be employed in the above polymers include:

- 2,2-bis-(4-hydroxyphenyl)propane (or bisphenol-A);
 - 2,4'-dihydroxydiphenyl methane;
 - bis-(2-hydroxyphenyl)methane;
 - bis-(4-hydroxyphenyl)methane;
 - bis-(4-hydroxy-5-nitrophenyl)methane;
 - bis-(4-hydroxy-2,6-dimethyl-3-methoxyphenyl)methane;
 - 1,1-bis-(4-hydroxyphenyl)ethane;
 - 1,2-bis-(4-hydroxyphenyl)ethane;
 - 1,1-bis-(4-hydroxy-2-chlorophenyl)ethane;
 - 1,1-bis-(2,5-dimethyl-4-hydroxyphenyl)ethane;
 - 1,3-bis-(3-methyl-4-hydroxyphenyl)propane;
 - 2,2-bis-(3-phenyl-4-hydroxyphenyl)propane;
 - 2,2-bis-(3-isopropyl-4-hydroxyphenyl)propane;
 - 2,2-bis-(4-hydroxyphenyl)propane;
 - 2,2-bis-(4-hydroxyphenyl)pentane;
 - 3,3-bis-(4-hydroxyphenyl)pentane;
 - 2,2-bis-(4-hydroxyphenyl)heptane;
 - bis-(4-hydroxyphenyl)phenylmethane;
 - bis-(4-hydroxyphenyl)cyclohexylmethane;
 - 1,2-bis-(4-hydroxyphenyl)-1,2-bis-(phenyl)propane;
 - 2,2-bis-(4-hydroxyphenyl)-1-phenylpropane; and the like.
- Also included are dihydroxybenzenes typified by hydroquinone and resorcinol; dihydroxybiphenyls such as 4,4'-dihydroxybiphenyl;

2,2'- dihydroxybiphenyl; 2,4'-dihydroxybiphenyl; dihydroxynaphthalenes such as 2,6-dihydroxynaphthalene.

Also useful are dihydric phenols wherein E is a sulfur-containing radical such as the dihydroxy aryl sulfones exemplified by bis-(4-hydroxyphenyl)sulfone; 2,4'-dihydroxydiphenyl sulfone; bis-(3,5-dimethyl-4-hydroxyphenyl)sulfone; 3-chloro-2,4'-dihydroxydiphenyl sulfone; 3-chloro-bis-(4-hydroxyphenyl)sulfone; and 4,4'-dihydroxytriphenyl disulfone.

The preparation of these and other useful sulfones are described in U.S. Patent 2,288,282. Hydroxy terminated polysulfones as well as substituted sulfones using halogen, nitrogen, alkyl radicals, are also useful.

Dihydroxy aromatic ethers such as those described in U.S. Patent 3,148,172 are useful as the dihydric phenol herein. The dihydroxy aromatic ethers may be prepared as described in U.S. Patent 2,739,171.

Illustrative of such compounds are the following:

4,4'-dihydroxydiphenyl ether;
4,4'-dihydroxytriphenyl ether;
the 4,3'-, 4,2'-, 4,1'-, 2,2'-, 2,3'-dihydroxydiphenyl ethers;
4,4'-dihydroxy-2,6-dimethyldiphenyl ether;
4,4'-dihydroxy-2,5-dimethyldiphenyl ether;
4,4'-dihydroxy-3,3'-diisobutyldiphenyl ether;
4,4'-dihydroxy-3,3'-diisopropyldiphenyl ether;
4,4'-dihydroxy-3,3'-dinitrodiphenyl ether;
4,4'-dihydroxy-3,3'-dichlorodiphenyl ether;
4,4'-dihydroxy-3,3'-difluorodiphenyl ether;
4,4'-dihydroxy-2,3'-dibromodiphenyl ether;
6,6'-dihydroxydinaphthyl-2,2'-ether;
6,6'-dihydroxy-5,5'-dichlorodinaphthyl-2,2'-ether;
4,4'-dihydroxypentaphenyl ether;
4,4'-dihydroxy-2,6-dimethoxydiphenyl ether; and
4,4'-dihydroxy-2,5-diethoxydiphenyl ether.

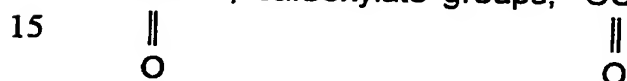
Mixtures of the dihydric phenols can also be employed, and where dihydric phenol is mentioned herein, mixtures of such materials are considered to be included. Other dihydric phenols which are suitable are disclosed in U.S. Pat. Nos. 2,999,835; 3,028,365; 3,334,154; 4,131,575.

The carbonate precursor used to produce the polycarbonate resins may be either a carbonyl halide, a carbonate ester, or a haloformate. Typical of

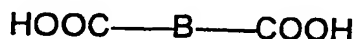
the carbonate esters are diphenyl carbonate, di(halophenyl)carbonates such as di(chlorophenyl)carbonate, di(bromophenyl)carbonate, di(trichlorophenyl)carbonate, di(tribromophenyl)carbonate, di(alkylphenyl)carbonate such as di(tolyl)carbonate, phenyltolyl carbonate, dichloronaphthyl chlorophenyl carbonate, and the like.

The haloformates suitable for use herein include bis-haloformates of dihydric phenols such as bis-chloroformates of hydroquinone, or glycols such as bis-haloformates of ethylene glycol, neopentyl glycol or polyethylene glycol. While other carbonate precursors will occur to those skilled in the art, carbonyl chloride, also known as phosgene, is preferred.

Included within the term "polycarbonates", for the purposes of this invention are the poly(ester-carbonate)resins. These resins may generally be described as polymers comprising recurring carbonate groups, -O-C-O-, carboxylate groups, -OC-, and aromatic carbocyclic groups in the



linear polymer chain, in which at least some of the carboxylate groups and at least some of the carbonate groups are bonded directly to ring carbon atoms of the aromatic carbocyclic groups. These poly(ester-carbonate)polymers, in general, are prepared by reacting an aromatic difunctional carboxylic acid or ester forming derivative, a dihydric phenol and a carbonate precursor. The preparation of poly(ester-carbonates) which may be employed in the compositions of the present invention is described in U.S. Patent Nos. 3,030,331; 3,169,121; 3,207,914; 4,194,038 and 4,156,069. The poly(ester-carbonates) which are preferred in the practice of the present invention include the aromatic poly(ester-carbonates) derived from dihydric phenols, aromatic dicarboxylic acids or their reactive ester forming derivatives such as the aromatic diacid halides, and phosgene. The aromatic difunctional carboxylic acids suitable for producing poly(ester-carbonates) may be represented by the general formula:



wherein B represents an aromatic radical such as phenylene, naphthalene, biphenylene, substituted phenylene; two or more aromatic groups connected through non-aromatic linkages such as those defined by E in formula I; or a

divalent aliphatic-aromatic hydrocarbon radical such as an aralkyl or alkaryl radical.

For purposes of the present invention, the aromatic dicarboxylic acids or their reactive derivatives such as, for example, the acid halides or diphenyl esters are preferred. Thus, in the preferred aromatic difunctional carboxylic acids, as represented by formula II, B is an aromatic radical such as phenylene, biphenylene, naphthalene, substituted phenylene, etc. . . Some non-limiting examples of some aromatic dicarboxylic acids which may be used in preparing the poly(ester-carbonate) of the instant invention include phthalic acid, isophthalic acid, terephthalic acid, o-, m-, and p-phenylenediacetic acid, and the polynuclear aromatic acids such as diphenyl dicarboxylic acids, and isomeric naphthalene dicarboxylic acids. The aromatics may be substituted with Y groups in the same manner as the formula I aromatics are substituted. Of course, these acids may be used individually or as mixtures of two or more different acids. A particularly useful class of aromatic poly(ester-carbonates) is that derived from bisphenol-A, isophthalic acid, terephthalic acid, or a mixture of isophthalic acid and terephthalic acid, or the reactive derivatives of these acids such as terephthaloyl dichloride, isophthaloyl dichloride, or a mixture of isophthaloyl dichloride and terephthaloyl dichloride, and phosgene. The molar proportion of ester units in the poly(ester-carbonate) is generally from about 2 to 90 mole percent and preferably about 3 to 80 mole percent. The molar range of terephthalate units, with the remainder of the copolymer ester units preferably comprising isophthalate units, is generally from about 2 to about 90 percent, and preferably from about 5 to about 50 percent.

Preferably, the polycarbonate compositions of the invention include less than 1% by weight of titanium oxide, and at best less than 0.1% by weight of titanium oxide. The most preferred polycarbonate compositions are free of titanium oxide.

Furthermore, the preferred polycarbonate compositions of the invention have light transmission values in the visible range of at least 80%, preferably of at least 85% and more preferably of about 90%.

The present invention is further illustrated by the following examples in which, unless otherwise stated, all percentages and parts are by weight.

1. POLYCARBONATES

Two different polycarbonates have been used. These two polymers are homopolymers of bisphenol-A polycarbonates which are identified below as Control PC and Hi-Flow PC.

5 Selected properties of these PC are given in table 1.

TABLE 1

<u>MATERIAL</u>	<u>Notched Izod</u> <u>Impact</u> Strength(J/m)	<u>M_w</u> (g/mole)	<u>M_n</u> (g/mole)	<u>Melt Flow</u> (g/10 min)
Control PC	830	41,000	17,000	10
Hi-Flow PC	750	37,000	15,000	19

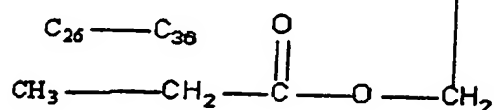
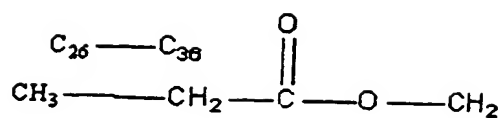
10 2. COMPOSITIONS 1 TO 6 AND COMPARATIVE COMPOSITIONS
A TO D

Compositions 1 to 6 according to the invention as well comparative compositions A to D have been prepared by blending different lubricants in various proportions with control PC as indicated in table 2 below:

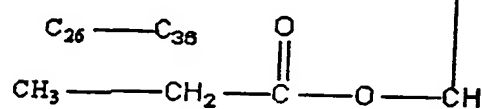
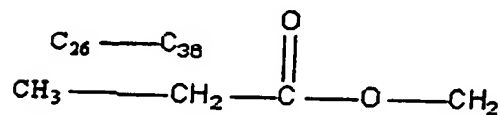
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TABLE 2

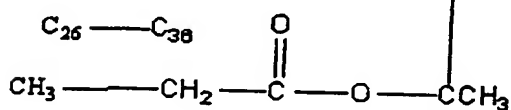
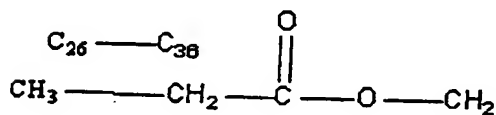
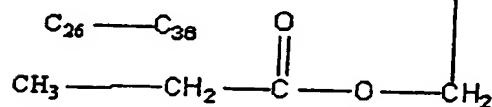
<u>Composition</u>	<u>Lubricant</u>	<u>% by weight</u>
1	L/MONTAN	0.5
2	L/MONTAN	1.5
3	M/MONTAN	0.5
4	M/ MONTAN	1.5
5	L/PETS	0.5
6	L/PETS	1.5
A	H/MONTAN	0.5
B	H/MONTAN	1.5
C	M/PETS	0.5
D	M/PETS	1.5



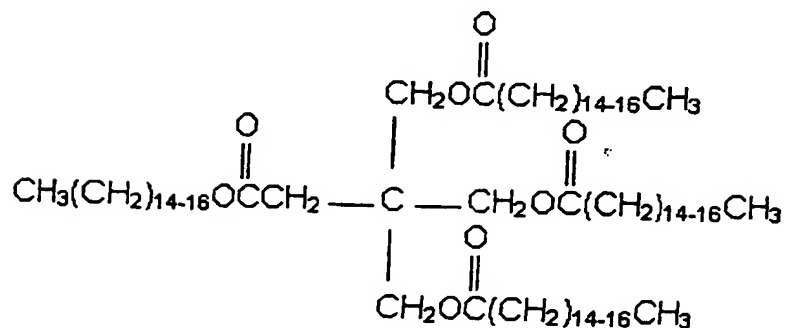
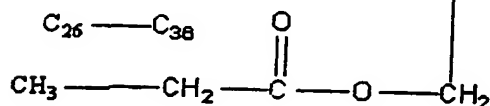
L/MONTAN



M/MONTAN



M/MONTAN



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3. COMPOUNDING

All compositions were compounded using a twin co-rotating screw extruder from Werner and Pfleiderer, model ZSK 30. The Control PC was dried at 121°C for 4 hours before pre-mixing. The Control PC was pre-mixed with each of the lubricants prior to compounding. The setup of the extruder was based on the polycarbonate operating temperature range, quality of extrudate and motor amperage. The temperatures were kept in the intermediate range of polycarbonate for preventing degradation. The feed rate was changed to maintain the amperage close to 70% of total load while maintaining the extrudate for pelletizing purposes. The motor amperage changed as different lubricants were compounded. The processing parameters are shown in table 3 below.

TABLE 3

Feed throat	Zone 2	Zone 3	Zone 4	Die	Screw speed
°C	°C	°C	°C	°C	rpm
182	260	315	315	300	130

4. RHEOLOGY

Viscosity was measured according to ASTM D3835 using a Goettfert capillary rheometer model rheograph 2003 with a 0.5 mm diameter capillary having an L/D of 20. The apparent viscosity of each composition as well as of the Control PC and Hi-Flow PC was measured over a shear rate range of 8,000 to 200 s⁻¹ at 282°C. The conditions for the rheometer included a preheating time of 5 minutes.

The apparent viscosities at a shear rate of 200 s⁻¹ and 282°C are given in table 4.

TABLE 4

<u>MATERIAL</u>	<u>APPARENT VISCOSITY</u> <u>(Pa.s)</u>
Control PC	794
Hi-Flow PC	440
Comparative A	641
Comparative B	373
Comparative C	635
Comparative D	660
Composition 1	537
Composition 2	262
Composition 3	415
Composition 4	220
Composition 5	702
Composition 6	232

5 Figure 1 is a graph of the apparent viscosities at a shear rate of 200 s^{-1} in function of the temperature for the Control PC, the Hi-Flow PC, compositions 1 and 3 according to the invention and comparative composition A.

Figure 2 is a graph similar to the graph of figure 1 for composition 6 and comparative composition D.

10 Figure 3 is a graph of the apparent viscosities at a shear rate of 200 s^{-1} and 282°C function of the lubricant concentration (% by weight) using as lubricant M/MONTAN, L/PETS and for comparison M/PETS.

15 The table as well as figures 1-3 show that the lubricants according to the invention greatly reduce the melt viscosity of PC compared to even structurally closely related compounds.

5. MOLDING AND IMPACT TESTING

Test samples for Notch Izod Impact test were molded using a ROBOSHOT 110R injection molding machine manufactured by CINCINNATI-

FANUC. The barrel capacity was 165.5 cm³ (PS). The processing parameters are given in table 5.

TABLE 5

5

<u>CONDITIONS</u>	<u>VALUE</u>
Nozzle (°C)	282
Front (°C)	285
Middle (°C)	282
Rear (°C)	280
Feed Throat (°C)	49
Injection velocity (mm/s)	31.75
Packing Pressure (MPa)	34.5
Packing Time (s)	18.0
Mold Temperature (°C)	107
Cooling Time (s)	40

While the above conditions were maintained, the peak pressured at the transfer point was monitored for each concentration. This value was collected over 20 shots and averaged to represent the peak pressure for the sample (given in table 6 below).

10

TABLE 6 (PEAK PRESSURES)

	Weight % additive					
	0	0.25	0.5	1	1.5	2
Control PC	123	123	123	123	123	123
Hi-Flow PC	94	94	94	94	94	94
M/MONTAN	123	90	82	70	60	52
L/PETS	123	120	108	78	69	61

15 **EXAMPLES 1 To 7 AND COMPARATIVE EXAMPLES A AND B**

Eight series of 10 plano lenses were injection molded as described above, starting from the compositions indicated in table 7 below.

The lenses have a 75 mm diameter and a 1.5 mm center thickness.

TABLE 7

<u>EXAMPLE N°</u>	<u>COMPOSITION</u>
Comparative A	Control PC
Comparative B	Hi-Flow PC
1	Composition 5
2	Composition 6
3	Control PC + 2.0 % wt L/PETS
4	Control PC + 0.25 % wt M/MONTAN
5	Composition 3
6	Control PC + 1.0% wt M/MONTAN
7	Control PC + 0.1 % wt M/MONTAN 0.5% wt L/PETS

Apparent viscosities for some of the compositions at two shear
 5 strengthes are given in table 8.

TABLE 8

<u>COMPOSITIONS</u>	<u>APPARENT VISCOSITY (Pa.s)</u>	
	200 s ⁻¹	4,000 s ⁻¹
Control PC	793	261
Hi-Flow PC	440	201
Example 1	617	237
Example 7	324	207

10 The lenses (10 lenses) of each serie were coated on both faces with a polysiloxane hard coat containing an epoxy silane and submitted to a high speed impact. According to this test, plano lenses are impacted at room temperature with a quater inch (6.35 mm) steel ball at a 6 m.s speed. The result
 15 was determined as a pass or fail depending on the sample, condition after the impact. Failure corresponds to the steel ball completely passing through the lens or the lens being broken into two or more pieces. The number of lenses which passes successfully the test in each serie was recorded. The results are given below in table 9.

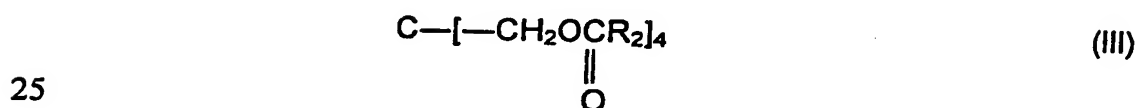
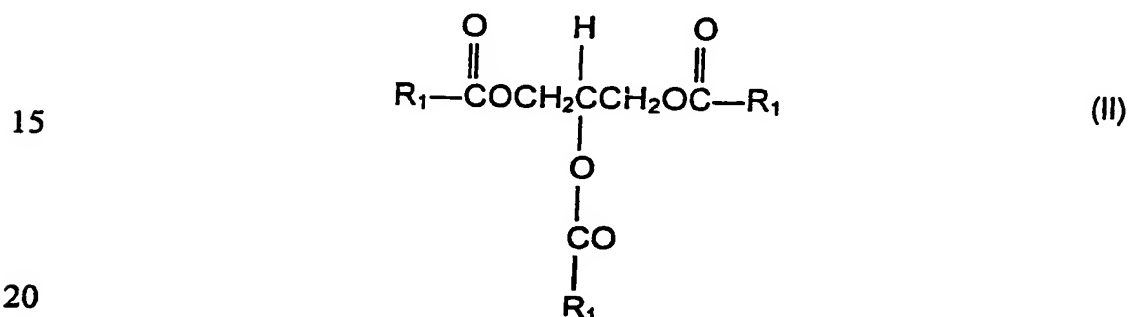
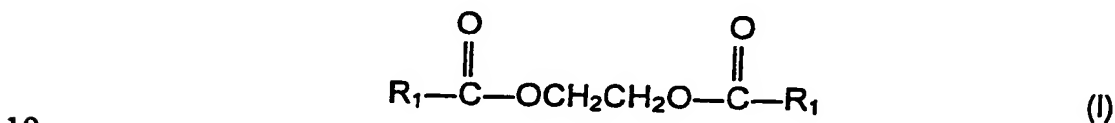
TABLE 9

<u>EXAMPLE N°</u>	<u>% PASS</u>
Comparative A	100
Comparative B	0
1	60
2	0
3	0
4	20
5	0
7	60

The polycarbonate compositions of the invention are particularly
5 useful for making optical and ophthalmic lenses.

CLAIMS

1. A polycarbonate composition which comprises an effective amount of at least one lubricant selected from the group consisting of the compounds of formulas:



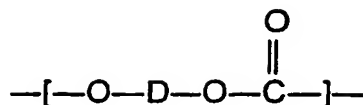
in which R_1 is a mixture of C_{28} - C_{38} linear alkyl groups and R_2 is a mixture of C_{15} - C_{17} linear alkyl groups, and mixtures of these compounds.

2. A composition according to claim 1, wherein the lubricant is a mixture of compounds of formula (II) and of formula (III).

3. A composition according to claim 1 or 2, wherein the amount of lubricant ranges from 0.01 to 5% by weight based on the total weight of polycarbonates present in the composition.

4. A composition according to claim 1 or 2, wherein the amount of lubricant ranges from 0.1 to 2.5% by weight based on the total weight of polycarbonates present in the composition.

5. A composition according to claim 1 or 2, wherein the polycarbonate has recurring structural units of the formula:



5

wherein D is a divalent aromatic radical of a dihydric phenol.

6. A composition according to claim 1 or 2, wherein the polycarbonate is a homopolycarbonate of bisphenol-A.

7. An optical or ophthalmic lens made of a polycarbonate
10 composition as set forth in claim 1.

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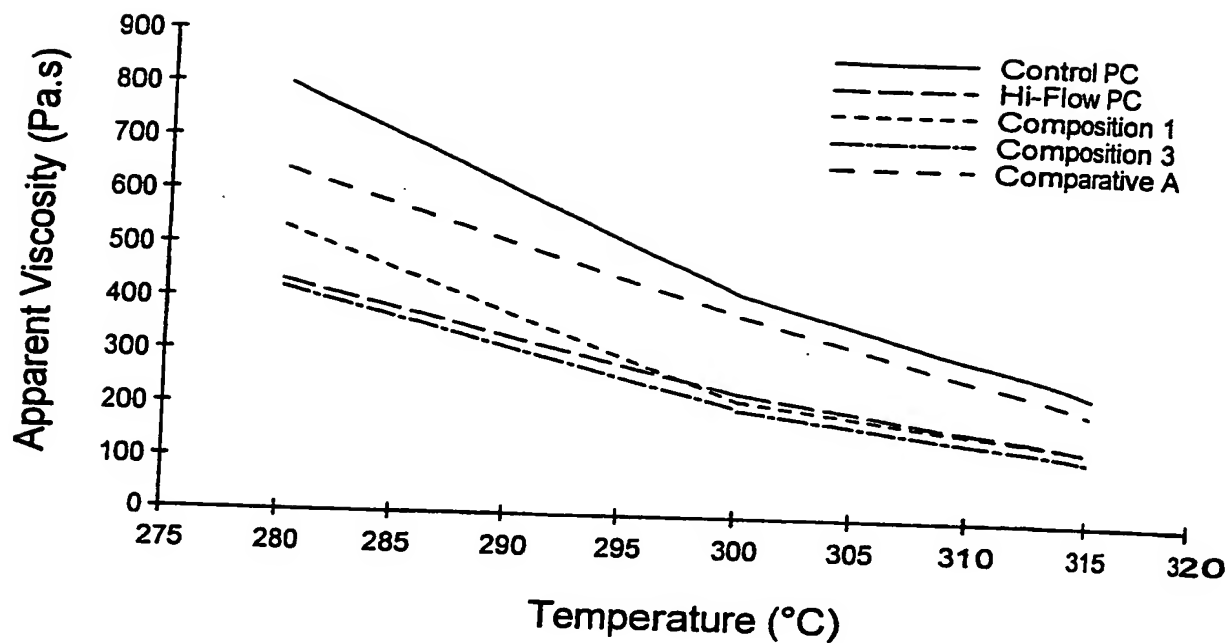


FIG. 1

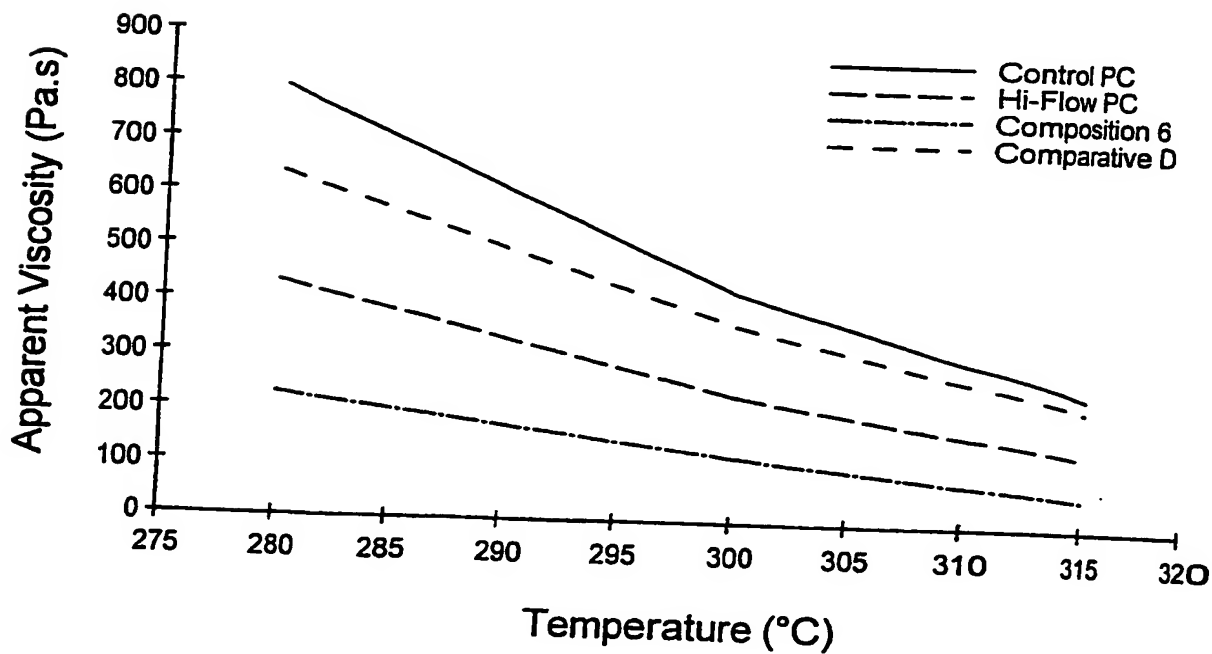


FIG. 2

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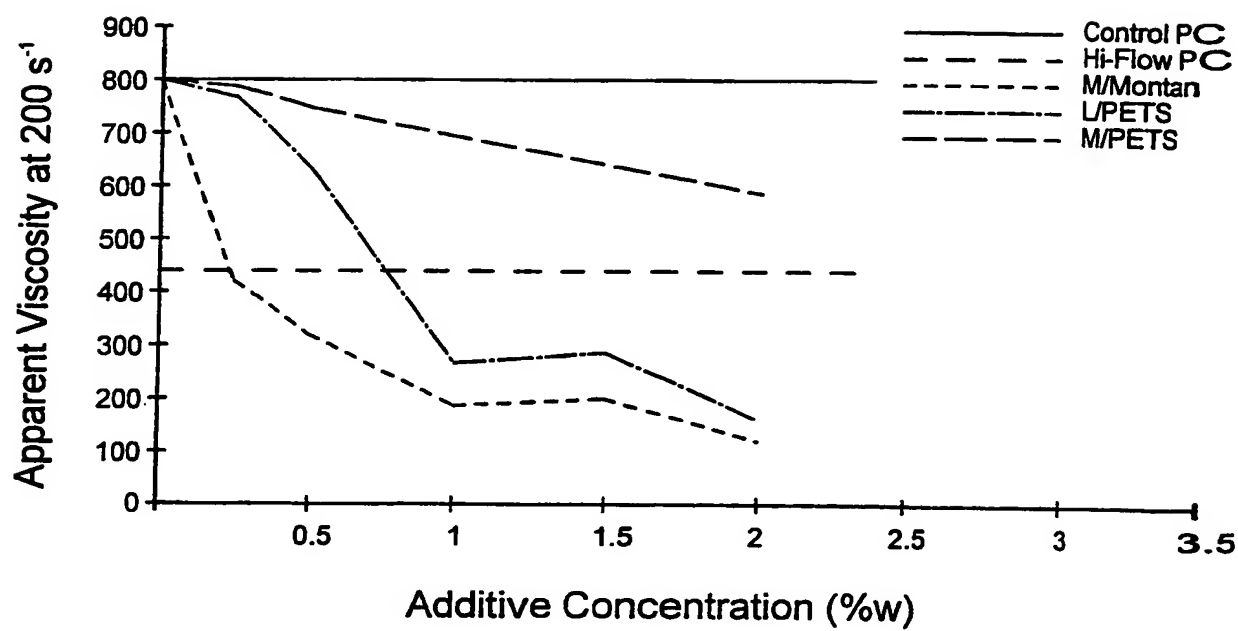


FIG. 3

INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 00/11150

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C08K5/103

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C08K C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EP0-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 596 034 A (KRISHNAN SIVARAM ET AL) 21 January 1997 (1997-01-21) claims 1,2; examples; table 1	1,3-6
X	US 4 097 435 A (RAWLINGS HERBERT L ET AL) 27 June 1978 (1978-06-27) column 4, line 33-55; examples 1,4; table 1	1,3-6
X	EP 0 511 640 A (TEIJIN CHEMICALS LTD) 4 November 1992 (1992-11-04) page 1, line 7; examples 1,COMP.3-4,9-10	1,3-7
A	examples 6,COMP,11	2
X,P	US 6 008 280 A (PISIPATI RAMESH M ET AL) 28 December 1999 (1999-12-28) column 3, line 50 column 6, line 5-20; claims 1,2	1,3-6

☐ Further documents are listed in the continuation of box C.

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Date of the actual completion of the international search

30 March 2001

Date of mailing of the international search report

12/04/2001

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Friederich, P

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 00/11150

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
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US 4097435	A	27-06-1978	CA 1098244 A DE 2814651 A FR 2387266 A GB 1579119 A IT 1102552 B JP 53126055 A	24-03-1981 12-10-1978 10-11-1978 12-11-1980 07-10-1985 02-11-1978
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US 6008280	A	28-12-1999	NONE	

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